Recommended practices for the calibration and use of capacitance diaphragm gages as transfer standards

Richard W. Hyland
National Institute of Standards and Technology, Gaithersburg, Maryland 20899

Richard L. Shaffer
EG&G Mound Applied Technologies, Miamisburg, Ohio 45343

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Capacitance diaphragm gages, which were previously known as capacitance manometers, have become a ubiquitous part of systems used in the vacuum community because of their ease of use, compatibility with most gases, and potential accuracy and resolution. They are used not only as pressure measuring devices, but also as transfer standards against which other pressure instruments are calibrated. There are a variety of precautions that must be considered when using these instruments. The American Vacuum Society is therefore presenting this Recommended Practice as a guide to their proper calibration and use as transfer standards. The document covers suggested plumbing configurations, laboratory practices, calibration methods, and reports.

CONTENTS

I. INTRODUCTORY COMMENTS

This document is intended to provide guidance for the calibration and use of capacitance diaphragm gages (CDGs) as transfer standards. Procedures will not be defined in such detail that a user has no choice of instrumentation or sequence of events. Rather, an overview of several commonly used calibration procedures will be given, and interested persons may use, adapt, modify, or improvise as they see fit.

The text has been prepared by a subcommittee of the Recommended Practices Committee of the American Vacuum Society. This presentation is to solicit comments from the manufacturer/user community before publication in final form. The members of the subcommittee were Randy Clark (MKS Instruments, Andover, MA), Robert Ferran (Ferran Scientific, Inc., San Diego, CA), Hollis L. Gray, Jr. (Edwards High Vacuum, Wayland, MA), Peter M. Hauser (Tylan General, Inc., San Diego, CA), Theodore Held (Abbott Labs, N. Chicago, IL), Richard W. Hyland (National Institute of Standards and Technology, Gaithersburg, MD), William B. Leisher (Sandia National Labs, Albuquerque NM), Paula B. Pokorny (EG&G Florida, Inc.), Richard Shaffer (EG&G Mound Applied Technologies, Miamisburg OH), and Duane H. Wright (Leybold Inficon, Inc., East Syracuse, NY).

II. SCOPE

This document gives general descriptions of and considerations necessary for calibration systems, and describes several methods for the calibration of CDGs against various standards.

At the time of the writing of this document these standards apply to the pressure range from about 10^{-5} Pa (10^{-7} Torr) to well above 10^{5} Pa (1 atm), covering the pressure range of currently available CDGs which are used for vacuum work. Also outlined is the documentation which should be considered part of the calibration procedure.

Note: Further details concerning subsequent sections of this document can be found in the Appendix. The appendix sections contain the same numbering and titles as the main text, although not all sections of the text are represented.

III. TERMINOLOGY

A. Conversion factors

Following are several commonly used pressure units and conversion factors.

$$1\ \text{Pa} = 0.007\,500\,62\ \text{Torr}$$
$$= 0.01\ \text{mbar} = 1.450\,377 \times 10^{-4}\ \text{psi},$$
$$1\ \text{Torr} = 133.3224\ \text{Pa}$$
$$= 1.333\,224\ \text{mbar} = 0.0193\,368\ \text{psi},$$
$$1\ \text{standard atm} = 760\ \text{Torr} = 101\,325\ \text{Pa}$$
$$= 1013.25\ \text{mbar} = 14.695\,95\ \text{psi},$$
$$1\ \text{psi} = 6894.757\ \text{Pa}$$
$$= 68.947\,57\ \text{mbar} = 51.714\,93\ \text{Torr}.$$

The pascal (Pa), defined as a newton per square meter, is the internationally sanctioned pressure unit.

The use of lengths of liquid columns as pressure units, e.g., inches of water, is strongly discouraged. These units are dimensionally incorrect, and generally misunderstood as soon as any degree of precision is required.

B. General

Additional definitions and terminology which apply to this document are found in the appendix. The AVS Dictionary of Terms[1] should be consulted for other terminology.

IV. CAPACITANCE DIAPHRAGM GAGES (CDGs)

The CDG pressure measuring system provides rapid pressure determinations over 4–6 pressure decades with an accuracy and stability that is considered acceptable by a wide spectrum of users of vacuum technology and equipment. CDGs have a small internal volume, high sensitivity, rapid response, can generally be used with a wide variety of gases, and are generally compatible with data acquisition systems. The long and short-term stabilities vary even among the same type, and they are affected by such things as temperature variations, position, and vibration. These environmental factors are considered below. As with any instrumentation, it must be decided whether these devices will meet the need, based on information from the manufacturers and other users.

The second section of the bibliography, Refs. 34–42, indicates some of the many situations where CDG's have been used for calibration, while Refs. 43–47 give general comments on calibration procedures and requirements.

A. General description

The capacitance diaphragm gage is a pressure measuring system consisting of a pressure sensor, electrical circuitry to convert the physical reaction of the sensor into an electrical signal, and signal conditioning circuitry to process this signal into an output which is the direct representation of pressure having any or all of the following forms:

(1) Electrical:

(a) analog, e.g., voltage, current,

(b) digital, e.g., IEEE 488, RS 232, etc.;

(2) Visual:

(a) analog, e.g., bar graphs, meters,

(b) digital, e.g., LED, LCD, CRT.

The CDG may embody a variety of operational features such as autoranging, autozeroing, and temperature control. It may be made up of modular components such as transducers, power supplies, and digital readouts.

The sensor is essentially a two sided cavity, separated by a nonporous diaphragm. Figures 1 and 2 show the commonly used configurations. The sensor may have vacuum permanently sealed on one side, and a port on the other to allow for absolute pressure measurements, or it may have ports on both sides of the cavity to allow for differential measurements. In the latter case the reference side must be continually evacuated for an absolute pressure measurement. The diaphragm forms the movable side of a variable capacitor. To construct the sensor, electrodes are added to one or both sides of the diaphragm to form the stationary plate(s) of the capacitor. See Refs. 2 and 3 for examples of additional construction details.

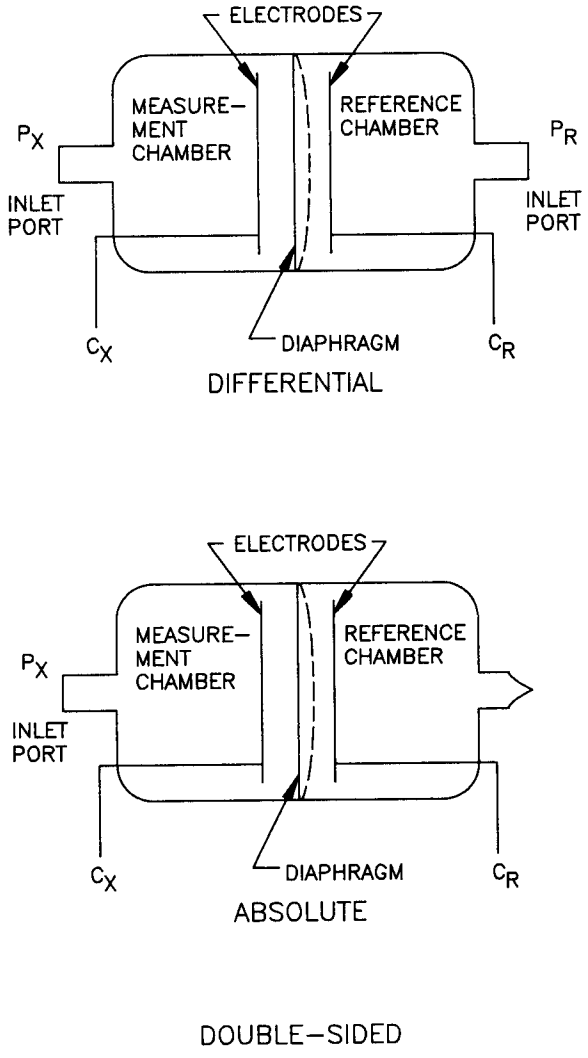


FIG. 1. Double-sided CDG sensors, differential and absolute. P_x and P_R are, respectively, the unknown and reference pressures, and C_x and C_R are the capacitor plates on the unknown and reference sides.

Some capacitance diaphragm transducer models can be operated not only at temperatures in excess of 300 °C but also below liquid nitrogen temperatures. The user should be aware that special considerations may be necessary for calibration and use at elevated or reduced temperatures. These include thermal transpiration (discussed later in this document), and temperature corrections. Consult the manufacturer for guidance.

In addition to a wide selection of ranges, various materials and types of construction are available, depending on the needs of the user. For example, the sensors in Figs. 1 and 2 may or may not be in a temperature controlled enclosure. See Refs. 2–6 for more details.

It is recognized that cost, complexity, and performance tradeoffs result in a wide variety of inherent uncertainties in commercial CDGs. Sometimes unintentional quality variations exist even within a group of the same type. It will not therefore be possible at any point in this document to give uncertainty statements which apply to all CDGs. References 7, 8, and 9 are examples of existing documentation of performance and uncertainties.

V. CONSIDERATIONS FOR CALIBRATION AND USE OF CDGs

The calibration techniques used for CDGs are varied, and depend in part on the capabilities of the particular unit. Several acceptable techniques will be described. The methods given are intended as examples. Whatever method is used, it is essential that a proper error analysis be performed.

Note: No calibration is worthy of the name if an intensive effort has not been made to locate and assess the magnitudes of the various contributing uncertainties.

The next several paragraphs and sections apply to any calibration. It is assumed, unless otherwise mentioned, that the calibrator is interested in getting the best performance from the particular CDG.

A. The calibration system

Several possible calibration systems are illustrated in the text. In all cases, there are precautions and factors that must be observed.

(a) It is important that the interconnecting plumbing between the pumps, standard, and instrument being calibrated has a large enough conductance to prevent pressure gradients that will affect the calibration. These gradients will arise from normal outgassing and small system leaks as well as from transient pressures if there are changes for example in dead-weight tester heights, pump speeds, or gas law (pres-

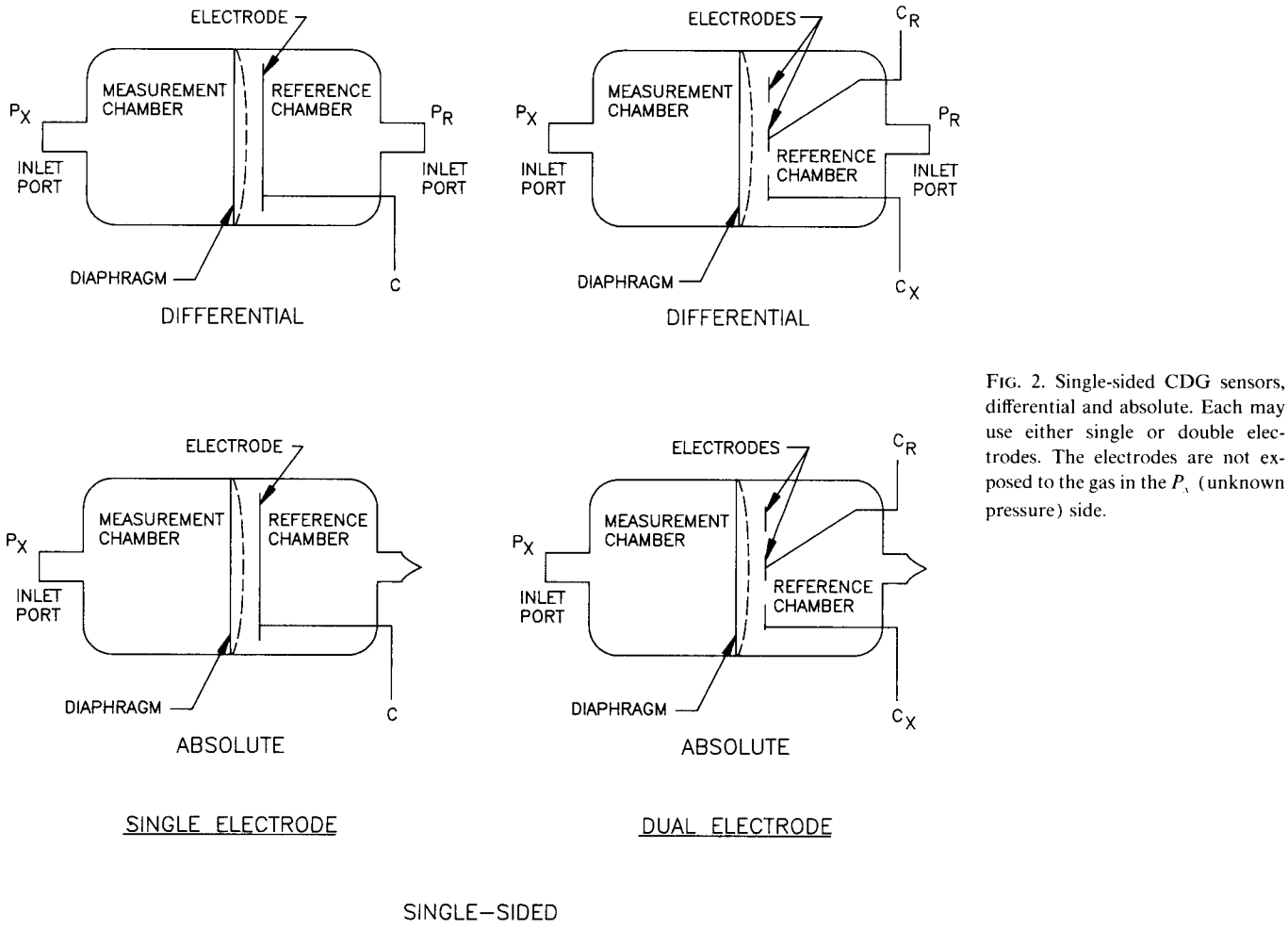


FIG. 2. Single-sided CDG sensors, differential and absolute. Each may use either single or double electrodes. The electrodes are not exposed to the gas in the P_X (unknown pressure) side.

sure–volume–temperature) effects. See Refs. 4–6, and 10 for other details.

(b) Some types of gradient effects are easily detected simply by closing valve or shutting off the pumps and observing changes in the outputs of the different gages on the system. This is also a good test of the overall leak integrity of the system.

(c) If absolute calibrations at pressures below about 0.1 Pa (10^{-3} Torr) are desired, for example, against a spinning rotor vacuum gage, it is strongly suggested that high vacuum techniques can be used in constructing and maintaining the system (See, e.g., Refs. 4–6, and 10).

(d) Whatever type of calibration system is used, it should be kept as clean and dry as possible. This means, for example, backfilling with dry gas to a pressure slightly above 1 atm before opening, and not leaving it exposed to atmospheric conditions any longer than necessary.

(e) Records should be kept of times required to evacuate to base pressure, and of any revisions or unusual events/observations. (see Sec. VII B 2.)

(f) Vacuum leaks and pressure leaks are not necessarily symmetric. If calibrations are to be performed using pressures above atmospheric, the system will have to be leak checked at the highest pressure to be used.

(g) Precautions should be taken to protect all gages against overpressure.

1. Personnel

Personnel performing calibrations should be familiar with the principle of operation and general characteristics of both CDGs and whatever device is being used as a standard, plus all ancillary equipment.

2. Environment

a. Temperature considerations. All CDGs respond in some degree to external temperature changes, as do the standards against which they are being calibrated. Although many CDGs have built-in heaters and sensors for temperature control, they all should be protected during both calibration and subsequent use from local air currents, such as those from air conditioning vents and cooling fans. If extreme temperature variations are encountered, it may be necessary to actively control the temperature of the immediate surroundings, even for CDGs with built-in temperature control. The degree to which the lack of temperature stability is a problem must be inferred from behavior in a particular environment.

If condensable gases are present in the system, care must be taken to avoid temperatures where liquid can form.

b. Cleanliness. Strict attention must be paid to maintaining cleanliness in order to insure leak-tight joints, low out-

gassing rates, and to prevent contamination of the instruments and/or the calibration stand and standard.

Regarding the possibility of contaminants, it is useful or perhaps even crucial to know the history of the CDG to be calibrated before placing it on the calibration stand, to prevent the transfer of such contaminants. See Ref. 11.

c. Stability of equipment. It is suggested that transfer standard and check gages (Sec. VII C 2) be kept on and evacuated when not in use. This keeps them in a relatively stable temperature environment, as well as unstressed, clean, and outgassed before the next use. All of these precautions will help prevent shifts in calibration and minimize the next start-up time.

3. Laboratory safety

Caution: Laboratory safety and the use of hazardous materials is beyond the scope of this document.

4. Certification of equipment

If applicable, all ancillary equipment (voltmeters, etc.), should be within their certification period and have known uncertainties which can be factored into the final uncertainty of the instrument being calibrated. Proper operation of computers and data gathering devices should be verified. Be aware that the addition of data-logging equipment can introduce errors such as noise, nonlinearity, and voltage offsets. If possible, one should perform all calibration procedures with such equipment in place.

Components of the same model of CDG pressure measuring systems may be interchangeable within the manufacturer's specifications. However, it is recommended that elements comprising the measurement system, including all ancillary equipment, be calibrated as a unit for the lowest uncertainty.

5. Base pressure considerations

In most vacuum calibration systems, "absolute" pressures are only "absolute" to the degree that the base pressure can be ignored. The generated pressures are always determined with respect to the base pressure.

The system base pressure can be ignored if it is insignificant relative to the desired uncertainty at the lowest calibration point. Otherwise, the base pressure would have to be known with an acceptable degree of uncertainty by some independent means. The base pressure can be no greater than the lowest desired calibration pressure. The base pressure can be determined for example with an ionization gage, thermocouple gage, or another CDG. The operation of any base pressure indicator must be thoroughly understood. Prior calibration is strongly urged.

Note that, if the base pressure is at least two orders of magnitude below that of the lowest desired calibration pressure, its contribution to that pressure is no more than 1%. This may be completely unimportant for many applications. However, if a measurement uncertainty of $\pm 0.5\%$ is required at the lowest calibration pressure (and no other error sources exist), the base pressure must be known to be two orders of magnitude less to within $\pm 50\%$.

Other examples are given in Sec. V D 1 b. The base pressure must be consistent with the user's requirements. In general, the lower the base pressure of the calibration system, the easier it will be to perform the calibrations.

6. Calibration interference considerations

There is always the possibility that a CDG, either in its role as a standard or as the device being calibrated, might interact with the device playing the opposite role, or with some other component (e.g., a source of electromagnetic noise) of the calibration system. A typical example of this would be a CDG on the opposite arm of a tee or cross from an ionization gage where the thermal radiation from the filament or grid can impinge directly on the internal cavity of the CDG. This has been shown to cause substantial errors in CDG calibrations.

There have been instances where the output voltage of one CDG signal conditioner has been affected by the proximity of another signal conditioner. The signal from one reference oscillator can beat against another (either through a ground loop or electromagnetic radiation), and cause oscillations in the output voltage. If this voltage is being used for control purposes, the consequences can be significant. This problem may be relieved by slightly changing the reference oscillator frequency. Contact the manufacturer for specific instructions.

7. Vibration

The transducers are sensitive to vibration. If there is an acceleration of the sensing element (the diaphragm) with a component along an axis perpendicular to the plane of the diaphragm, a force will be exerted which is indistinguishable from pressure. Shock or vibration will cause an output proportional to the level of those frequency components lying within the bandwidth of the CDG signal processing system (usually below 100 Hz).

Typically, the pressure indication caused by acceleration is of the order of 1 Pa per "g" ($g = 9.80_7$ m/s^2) for 1333 Pa full scale CDGs or below, and for higher range units on the order of 5 Pa per g of input along an axis perpendicular to the diaphragm. The effects can be minimized by proper orientation of the diaphragm relative to the principle axis of vibration, by concentrating on isolating sources of vibration (e.g., pumps) of the system to which the CDG is attached, by mounting the CDG on vibration isolators (rubber or cork sheets), and by adding mass to the system. If vibration cannot be adequately controlled by the above methods, additional filtering of the output signal can be added, at the expense of frequency response.

B. Connecting to the system

Connect the transducer to the calibration system, following the manufacturer's instructions and any requirements of the calibrating organization.

1. Leak testing

After connecting the standard and devices to be calibrated to the calibration system, the system should be evacuated

and checked for leaks by an appropriate method (e.g., pressure rise, helium leak detector). In addition to preventing the system from reaching its required base pressure, leaks may cause pressure differentials which can lead to calibration errors.

2. Port choice

Absolute CDGs have only one port for pressure application. Differential CDGs have a pressure port and a second port for the reference pressure. (See Figs. 1 and 2.) Since many differential CDGs are linearized only for positive differential pressures, the pressure should be applied from the correct direction. It will not, in general, damage a differential CDG to pressurize it in the reverse direction, but the behavior may not be optimum, and an existing calibration for pressurization in one direction may not be valid for the other. Consult the manufacturer for details.

3. Position

Another force acting upon the diaphragm is that of the gravitational field of the earth. For example, mounting the unit with the plane of the diaphragm parallel with the surface of the earth will produce a one g deflecting force, whereas mounting it with the plane perpendicular produces no deflecting force. The result is that the output between the two positions will be different.

In general, there is no problem if the transducer is rotated about an axis perpendicular to the plane of the diaphragm, as this does not alter the plane of the diaphragm relative to gravitational forces. It is prudent to mount the unit in the same orientation during calibration and use.

It is suggested that the transducer be mounted in such a way to prevent particles from falling into the diaphragm volume. For example, the pressure port of an absolute transducer should face either horizontal or downwards unless the manufacturer recommends otherwise.

Note: Sensitivity is not affected by the diaphragm orientation.

4. Support

Some transducers are not meant to be attached to the system or operated without some form of support. In particular, these should not be suspended by their own tubing. Consult the manufacturer's instructions if unsupported operation is necessary.

C. Warm-up

Note: Warm-up should occur under vacuum in order to minimize elastic recovery efforts, outgassing time, and temperature changes due to pump-down prior to calibration.

For CDGs with no active temperature regulation which operate at or near room temperature, the time allowed for warm-up may be minutes to hours. This will depend in large part on the temperature sensitivity of the gage (consult the manufacturer) and the stability of the ambient temperature.

For temperature regulated CDGs, equilibration times of the order of 4–8 h are often suggested. For the highest precision instruments operating in laboratories controlled to within $\pm$ 1 °C, this document recommends that, in the absence of manufacturer's instructions for a longer time period, a 20 h period be allowed.

Warm-up includes any and all ancillary equipment in the calibration area. Monitor the zero drift of the CDG as a function of time until its stability is satisfactory.

D. Initial adjustments

Whether or not any adjustments are to be made to the electronics (e.g., setting test points to certain values) before the calibration is a policy decision of the calibration laboratory (see the Appendix for additional discussion).

It is the policy of the facility to reset parameters to the manufacturer's original specifications, it may also be the policy to perform an "as-received" calibration beforehand. Check to be sure all required data have been gathered before making any changes.

Follow the manufacturer's procedures for making the adjustments, plus any that may be required by your facility. All unusual adjustments should become a part of the documented history of the device.

1. Zeroing

Zeroing is to be done after the warm-up period and generally consists of either zeroing both the signal conditioner and the sensor output or zeroing the sensor output alone. The manufacturer's instructions should be followed. The following configurations are typical:

(i) CDGs with single zero adjustment,

(ii) CDGs with multiple zero adjustments,

(iii) CDGs with multiple zero adjustments, and signal conditioner zero adjustment.

With the latter type CDGs there are additional adjustments, which vary from manufacturer to manufacturer, related to the sensitivity. Instruction manuals should be consulted.

a. Signal conditioner zeroing. If applicable, the electronics that controls the sensor and ancillary equipment must be set to read zero and full scale when the corresponding voltages are applied by the front panel selector switch. Generally, these adjustments and corresponding instructions are available for commercial devices. If the laboratory performing the calibration is using its own electronics for power and/or readout and control, provisions should be made for these adjustments.

Important: If the CDG has multiple ranges, zeroing on one range may not set the zero for another.

Upon completing the zeroing procedures, record zeros on all ranges. If there are significant changes in zero as one goes from one range to another, these offsets may be used to correct the data on different ranges. Some laboratories use only the zero offset determined for the most sensitive range, and make no further zero adjustments as the calibration proceeds to different ranges. Shifts are then accounted for during data reduction, and the results appear in the calibration report.

If only one range is to be used, then the zero should be adjusted for that range.

b. Zeroing absolute CDGs. Two ways to establish the zero of absolute CDGs are suggested.

(1) The preferred method is to establish a base pressure which, when its uncertainty is considered, is low enough to be an insignificant percentage of the lowest calibration pressure. For example, if the base pressure is known only to within an order of magnitude at 10^{-3} Pa, then one can only be sure it is less than 10^{-2} Pa. If the first calibration point is at 1 Pa, then this base pressure and its uncertainty can at most contribute a 1% uncertainty to the first point. As a second example, suppose the base pressure is known to be 10^{-2} Pa to within 1%. Then the uncertainty contributed by the base pressure at a calibration point of 10^{-2} Pa is only 1%. See the Appendix and Sec. V A 5.

(2) A pressure, known by other means (low compared to the maximum pressure of interest), is established within the calibration system, and the CDG to be calibrated is set to indicate that pressure, accounting for thermal transpiration effects and height difference (pressure head) effects if applicable.

Note: It may also be necessary to make an additional adjustment to the data. This will be discussed in Sec. V F 2 of the appendix.

c. Zeroing differential CDGs. Using clean metal tubing and as short a path as possible, connect the CDG pressure ports together and evacuate to the desired reference pressure. An arrangement such as that of Fig. 3, incorporating a by-pass valve B and valve C is suggested. Evacuate the system by opening valves B and C. Then close C and wait for a sufficiently stable output and set the zero.

For some applications, it may be necessary to zero the CDG with the measurement and reference ports attached to different vacuum sources. That is permissible as long as all devices associated with the calibration are zeroed in a consistent manner. Adjust the electronics so that the CDG reads either zero or a known differential pressure.

Note 1: Even with zero-applied differential pressure, the zero indication may be a function of line pressure (this is a necessary and easy check to make). Therefore, it is important to perform the zeroing procedure near the line pressure of interest. Do not, for example, set zero with the applied zero differential pressure at one atmosphere, then evacuate to some other zero differential pressure and assume the zero is unchanged.

Note 2: It is often assumed that a good zero can be obtained just by leaving both ports of the unit open to atmosphere. One will generally find that there is a large zero instability, because the gage is so sensitive that it responds to drafts in the room which can produce differential pressures across the short distance between ports. The ports must be isolated from these variations, preferably by interconnecting them with hard tubing. See Note 1.

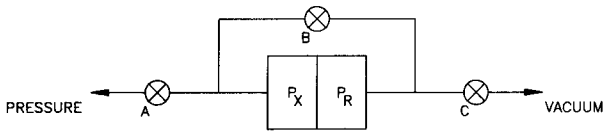


FIG. 3. Valve configuration for zeroing a differential CDG at vacuum. Close A and open C and the bypass valve B. Zeros can be achieved at any other pressure, using A and C to adjust.

E. Precycling

CDGs depend on the mechanical deflection of a metal membrane, and are therefore subject to hysteresis effects. In most cases these effects can be ignored. However, after warm-up, it is recommended that, at a minimum, the CDG be cycled from zero to full scale and back. This will help insure a common initial diaphragm condition for each use. Additionally, the CDG may benefit from cycling to atmospheric pressure. Consult manufacturer for guidance.

Precycling is particularly important for differential CDGs since there is no way of knowing where they may be on their hysteresis loop before use. It also permits a check of the overall system behavior before the actual calibration.

Cycling as described above or even during ordinary use introduces stresses in the sensor leading to a zero offset that can take hours to dissipate. Such zero effects can be of the order of 0.01% of full scale. They may diminish with time, or may remain at the 0.01% of full-scale level. In order to have repeatable low pressure (relative to the full scale of the particular CDG) data, be sure that enough time is allowed for the system to return to a stable zero before calibration.

Note: Cycling stress is one reason why, either during a calibration or actual use, it is recommended that data be taken on the low pressure range first.

1. CDG over-ranging or over-pressuring

Absolute CDGs are made to withstand an applied absolute pressure of at least 10^5 Pa (1 atm) without harm. Consult the manufacturer's literature. In order to achieve the best accuracy it is suggested that absolute CDGs with full scale ranges less than a 133 kPa be placed behind a permanent valve, which is closed when the calibration system is cycled beyond the range of the CDG.

Differential CDGs operating with a vacuum reference should receive the same considerations. Care should be taken to disconnect the measurement port first in order to avoid reverse overpressure.

F. The calibration gas

The calibration gas should be compatible with the vacuum system and end use of the CDG. If it is necessary to correct for height differences between the standard and the gage under calibration (See Sec. V F 3), the mean molecular weight of the gas must be known. For thermal transpiration calculations, the mean molecular diameter must be known (see Sec. V F 2).

1. Dielectric considerations

In single or double-sided CDGs where the gas is in contact with the electrodes, the gas becomes part of the dielectric

medium. The capacitance and therefore the CDG performance are dependent on the dielectric constant and ionizing properties of the calibration gas.

The gas used for calibration should have, within the accuracy limits required by the calibration, the same dielectric constant as that to be used in the calibration. For pressures near 1 atm, the commonly used calibration gases have nearly the same value for the dielectric constant. For examples: air—1.000 59, nitrogen—1.000 58, hydrogen—1.000 264, carbon dioxide—1.000 985.[12] The dielectric constant ϵ minus 1 is proportional to absolute pressure, i.e.,

$$\epsilon - 1 = KP, \tag{1}$$

so that considerations of calibration effects arising from dielectric constant variations become less important with decreasing absolute pressure ranges. However, if a differential CDG is calibrated at a high line pressure (see Sec. VI C, calibration with dead-weight testers) then subsequently pumped for use at vacuum, consideration must be given to the effect of the resulting change in dielectric constant on the calibration. Even in the case of a single-sided, dual electrode CDG (see Fig. 2) where the dielectric change affects both capacitor plates, the changes will not be exactly the same. This can cause second-order calibration shifts.

There can be extreme circumstances. For highly ionizing gases such as tritium, free electrons can cause a substantial change in the effective impedance of the capacitors at most pressures above a few tenths of a pascal. Do not calibrate with nitrogen, and then use that same calibration with a highly ionizing gas. See Ref. 13.

2. Thermal transpiration

Thermal transpiration is frequently a cause of calibration errors. The phenomenon occurs where the mean free path becomes of the order of the internal diameter of the interconnecting tubing between two volumes at different temperatures. In that case, the higher-temperature volume will be at higher pressure than the lower-temperature one. Such a condition can exist in the application of CDGs or their calibration.

For example, for a 0.42 cm i.d. tube connecting the CDG to a chamber with a temperature differential of 22 °C across that tubing, noticeable effects appear for pressures of the order of 100 Pa or less. The effect increases to a limit so that at pressures of the order of 0.1 Pa or less, the hot-to-cold pressure ratio, P_h/P_c, is given by

$$P_h/P_c = (T_h/T_c)^{1/2}. \tag{2}$$

Under these conditions a heated transducer operating at 45 °C and connected to a chamber at 23 °C will indicate a chamber pressure which is too high by a factor of $(318.15\ \text{K}/296.15\ \text{K})^{1/2}$ or 1.036, i.e., by 3.6%. See Refs. 14–17 for further information.

Note that thermal transpiration affects both absolute and differential transducers, and that it has nothing to do with the ideal gas law temperature effect.

Appendix E gives the complete form of the thermal transpiration equation and discusses a variety of factors which contribute to the effect, such as the gas species.

3. Height difference (pressure head) considerations

If two pressure devices are connected together with a height difference between them, the lower device will read a pressure which is greater than that of the uppermost device. If the height difference (in meters) is h, then the pressure difference in pascals is given by

$$dP = \rho g h, \tag{3}$$

where ρ is the density in kg/m^3, and g is the local value of gravity in m/s^2.

Since the (ideal gas) density can be calculated from

$$\rho = mP/RT, \tag{4}$$

where m is the molecular weight in kg/mol (e.g., for nitrogen, $m = 0.0280\ 13$), R is the gas constant (8.314 41 J/mol K), and T is the absolute temperature in kelvin, it follows that the fractional pressure variation with height is given by

$$\frac{dP}{P} = \left(\frac{mg}{RT}\right)h. \tag{5}$$

For room-temperature nitrogen, the correction amounts to about 116 parts per million (ppm) difference in the pressure for every meter difference in height, regardless of the pressure. Failure to account for pressure differences arising from height differences can be significant. It is surprising how often this correction is overlooked.

Clearly the standard and device being calibrated should be reasonably close in height to avoid any possibility of significant error.

VI. THE CALIBRATION

Assuming that all of the set-up, warm-up, and zeroing procedures discussed above have been followed, for both the transfer standard and the device to be calibrated, the calibration consists of applying pressure, waiting an appropriate time for thermal equilibrium to be achieved, and recording the indications of the standard and device under calibration as close to simultaneously as possible. The equilibration time after applying a new pressure will be a function of the calibration method and system volumes. At each point, the operator must watch the comparative indications of standard and device being calibrated. Once they start tracking each other, it can be assumed that the system has equilibrated. Experience will quickly teach the necessary waiting times, which will then be documented for the next operator.

Many CDGs have provision for checking the zero and full-scale indications of the electronics and auxiliary data acquisition equipment. For best accuracy, these can and should be checked occasionally during calibration. This procedure is not the same as zeroing the transducer indication, and therefore does not involve resetting the system to zero pressure. Consult the manufacturer's instruction manual for further information.

Reference 7 gives additional information on calibration techniques, including those discussed below.

A. Data order

The calibration should proceed from the lowest to the highest pressures, and then return, with the final points be-

ing again at zero. Because of diaphragm stresses (see Sec. V E) and the possible attendant zero shifts, the data taken during the return to lower pressures may show increasingly greater offsets from the data taken with increasing pressure, even if long pauses are made between data points. Should this occur, a decision will have to be made whether or not to include data obtained below a pressure where the offsets exceed acceptable uncertainty limits. If this is done, then the instrument must be used in a manner consistent with this calibration—i.e., do not use CDG for determining descending pressures below the limit specified in the calibration report. If the CDG is routinely used to cover the full range of increasing and decreasing pressures, then provision for any differences in ascending and descending pressures observed during calibration must be a part of the uncertainty statement.

It may also be necessary to decide whether observed differences between the ascending and descending data arise from the effect of hysteresis or from a zero offset, but this decision can usually be made based on the pattern of the offsets (hysteresis will show as larger offsets near mid-range, diminishing as the high and low ends are approached) as well as the actual offsets between the initial and final indicated zeros.

When CDG zero drifts are significant it may be beneficial to return to zero between each data point. Establish a pressure, return to zero and take the difference between the two readings.

B. General comments on data acquisition

Data should be taken at pressure intervals determined by the individual needs. If no satisfactory procedure has already been established, it is recommended that the calibration consist of no less than ten equally spaced points per decade on each scale of interest.

Enough readings should be taken at each setting to determine whether there is a random variation about the mean pressure. Readings should be taken to allow the calculation of an average value whose standard deviation is of an acceptable magnitude. See Ref. 18.

Many devices have a range switch which provides different sensitivities for different decades of pressure. At or near the pressure where a range change is to be made, take data on both ranges. There may be a significant (tenths of a percent) discontinuity in the pressure indication when the range is changed. Such discontinuities must be dealt with in the data reduction. See Sec. VII C and V D 1 for additional comments.

C. Calibrations against standards

This section will deal with the calibration of CDGs. Methods using spinning rotor gages, liquid manometers, deadweight testers, or other CDGs as standards be addressed. As stated at the beginning of this document, other devices or methods such as expansion systems are not precluded. Examples are found in Refs. 7 and 19–21. Some of the techniques require much attention to detail. The user must assess whether those types of calibrations are worth the effort for other than stable, high-resolution CDGs.

All set-up considerations and all precautions outlined above should be observed.

1. Calibrations against dead-weight testers (DWTs)

At the time of this writing, commercially available deadweight testers have accuracy capabilities of the order of 50 parts per million or better. They are inherently differential devices which, if the reference side is evacuated, can then be used for calibrating at absolute pressures as low as 2 kPa. If one DWT is balanced against another, pressure differentials much less than ten pascals can be generated at an absolute pressure of 2 kPa. DWTs therefore can be used to calibrate differential CDGs which can then be vacuum pumped to provide absolute measurements. If the CDG is operated at other than the temperature of the volume in which the pressure is to be determined, then thermal transpiration must be accounted for. Thermal transpiration is discussed at various places in this document. See Refs. 7, 22, and 23 for discussions of DWTs and their use in calibrating CDGs.

The DWT generates a pressure difference between the pressure above and below the piston. Some manufacturers provide a bell jar which can be placed over the piston-cylinder assembly and associated masses, allowing evacuation of the bell jar volume. In that case, the DWT becomes an absolute pressure generator. See Fig. 4(a). The lowest pressure is generated by the combined mass of the piston and weight carrier; higher pressures are generated by adding mass to the carrier. Absolute gages which do not need a calibration point below 2 kPa can use this technique directly.

To achieve lower differential pressures, two DWTs can be operated in opposition, i.e., one connected to one side of a differential CDG and the other connected to the other side [see Fig. 4(b)]. Thus the pressures generated by the weights of the carriers are canceled differentially. Small weights added to the carrier of one DWT can be used to generate small differential pressures. Using a two dead-weight tester system [see Ref. 7 and Fig. 4(b)], one can generate differential pressures of the order of ten Pascal, relative to the reference pressure. A differential CDG calibrated by this technique can then be used as an absolute standard by evacuating the reference side, provided proper consideration is given to thermal transpiration effects. Generally thermal transpiration will be negligible at pressures where calibrations versus DWTs are performed, but when a heated differential CDG is subsequently used in the absolute mode, that will no longer be true. The calculations of Appendix E should be used to estimate the corrections. Literature[17] indicates that the calculations are not precise over several pressure decades, depending on the system configuration. Allowance must be made for such variations in the error analysis.

It will be assumed that the DWTs have acceptable, documented calibrations on their effective areas and on the masses used to generate the pressures.

The piston-cylinder assembly should be cleaned and carefully leveled before use. By experience, the operators will learn to recognize problems by the feel of the piston in the

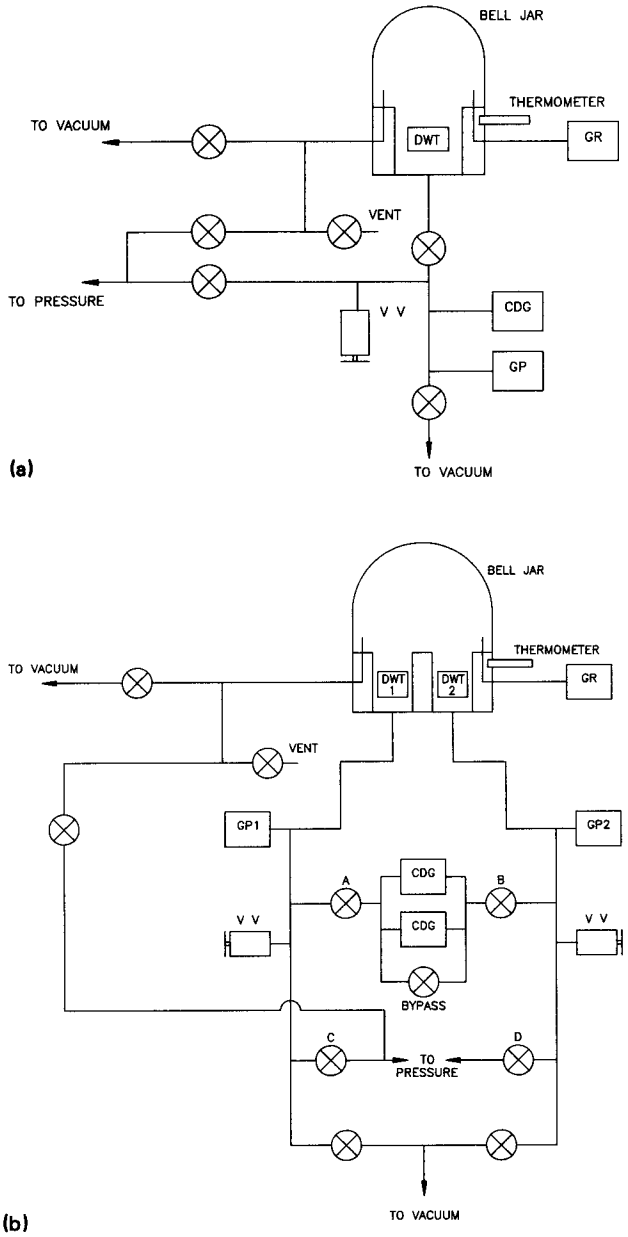


FIG. 4. System for calibrations of absolute CDGs against a DWT. GR and GP are gages for monitoring the reference and pressure sides of the system. VV is a variable volume to make small pressure adjustments. The bell jar can be vented or bled to atmospheric pressure with the test gas. Weights (pressures) of the DWT can be changed without disturbing the previous pressure in the CDG. *Note*: Not all DWTs are capable of absolute mode operation. (b) System for calibrating differential CDGs using two DWTs. This is two of the systems of (a) in parallel. Absolute gages could also be calibrated. DWTs enclosed in separate bell jars could be used with appropriate modifications.

cylinder when a gentle rotation is done by hand and by the length of time the piston rotates with a given loading. Manufacturer's instructions should be consulted for operating details.

a. The calibration system. Refer to Fig. 4(a) for a possible absolute calibration set-up and to 4(b) for a possible differential calibration set-up. The differential set-up consists of two absolute systems in opposition, with the addition of a bypass valve for zeroing the CDGs. Each system controlling the pressure of a DWT has the following components: a pressure gage for sensing pressure as the system is filled or evacuated (this need not be a calibrated gage); a variable volume valve or other method of making fine adjustments to the pressure; an associated pressure supply which can be shut off; a fine bleed gas supply (not shown in Fig. 4) to compensate for gas lost through the annulus between the piston and cylinder, and access to a vacuum system with the usual provisions for roughing and pumping. The bleed valve feature may not be necessary, depending on the length of time for which it is desired to hold the pressure constant. However, since the volume changer perturbs the system when used, the bleed gas is a feature to consider.

The reference sides of the systems have provisions for venting to atmosphere and pumping, as well as a gage to measure the reference pressure. The reference pressure within the bell jar must be added to the pressure generated by the weights. For highest accuracy measurements, a calibrated gage should be connected directly to the bell jar.

Figure 4(b) shows both dead-weight testers under the same bell jar. This is the preferred arrangement. However, they can be completely independent but in that case the reference pressures in the respective bell jars must be carefully measured and added to the pressure generated by the associated DWT.

Measure the temperature of each piston gage base to provide the information to correct the piston gage area to its reference temperature. Buoyancy corrections must be applied to all masses based on the bell jar pressure.

The pressures to be generated by the DWTs are selected by the choice of masses. These can be tailored to the needs of the particular laboratory.

b. Absolute calibration using a single DWT. Refer to Fig. 4(a). To begin the calibration, load the masses necessary to establish the first pressure. Evacuate the reference side of the system. When a satisfactory reference pressure has been obtained, raise the pressure under the piston until it begins to float. Spin the piston. Wait for stable pressure. Read the CDG, the piston gage temperature and the reference pressure. Repeat as required.

c. Differential calibration using two DWTs. Refer to Fig. 4(b). Even when using atmospheric pressure as the reference pressure for the DWTs, it is a good idea to place a bell jar over them to shield them from pressure variations.

(1) *Near atmospheric pressure*: For low differential pressure calibrations, two DWTs will be required. When calibrating with atmospheric pressure as the reference pressure for each DWT, one DWT would be used to generate the reference pressure for the CDGs. That is not the same as atmospheric pressure, but will be the sum of atmospheric pressure plus the pressure generated by the mass (probably of only the piston plus the weight carrier) on the DWT. The zero for the calibration should be taken at the CDG reference pressure. This is obtained by opening valve A, B, C, D, and the bypass. The two DWTs will each be loaded with the necessary masses required to generate the desired CDG reference (zero) pressure. Apply sufficient pressure to float the rotating DWTs. Once the CDG zeros are obtained, close the bypass valve.

Increment the weights on one of the DWTs to produce a differential pressure and take data as in Sec. VI C 1 b.

A CDG calibrated by the above procedure can be used as a vacuum standard by evacuating the reference side, providing that thermal transpiration effects, which do not appear during the DWT calibration, are accounted for (see comments in Sec. VI C 1). (See note 1, Sec. V D 1 c.)

Note: A calibrated differential CDG may have noticeable sensitivity errors associated with the change in reference line pressure. In order to improve the calibration accuracy using this technique, see below.

(2) *At reduced pressure*: Line pressure effects will be minimized by performing the calibration relative to pressures as close to the line pressure of interest as possible. This requires that the DWTs be capable of absolute mode operation. The calibration would proceed as in Sec. VI C 1 c 1, using just the weight carriers and the weights necessary to produce the differential pressure, except that the bell jar(s) will be evacuated and the magnitude of the reference pressure will have to be recorded. The result is a differential CDG calibrated relative to an absolute pressure determined by the mass on the reference-side DWT (2 kPa is the lowest available at this writing) plus the pressure in the bell jar. Note that the calibration will be performed relative to a line pressure where thermal transpiration is not important (2 kPa absolute or greater). If the calibrated CDG is subsequently used at pressures where thermal transpiration is a factor, its effects must be accounted for. See Sec. V F 2 in the text and Appendix E.

(3) *Extrapolating to zero line pressure*: Assume a CDG has been calibrated as described either in Sec. VI C 1 c 1 or Sec. VI C 1 c 2., and it is then desired to use it relative to a much lower (essentially zero) pressure.

If procedure Sec. VI C 1 c 2 has been performed at two or more different line pressures, the effect of line pressure can be extrapolated to "zero" pressure. This calculation requires accurate knowledge of the line pressures used. The calibration of the CDG can then be corrected from the calibration line pressure to "zero" line pressure. It is expected that this correction will be a small percentage of any pressure. As discussed in above sections, thermal transpiration must also be accounted for if heated transducers are being considered, because the extrapolations are probably being made from regions where thermal transpiration is negligible.

2. Calibrations using a calibrated CDG as a transfer standard

All comments of Secs. V and VI–VI B apply. It is assumed that the transfer standard has an acceptable calibration and is in good working condition. All corrections given in its calibration report must be applied or otherwise be accounted for. Thermal transpiration effects (see Sec. V F 2) should be accounted for in heated CDGs.

If the CDG is to be calibrated only at pressures above 10 Pa, or pressures at which sorption and desorption within the vacuum system are no longer significant, then a static system as shown in Fig. 5 will suffice. Shown are two CDGs, one absolute, one differential. Either one can be the standard. The ion gage shown is intended only as a base pressure indicator.

The system is evacuated to its base pressure, the vacuum pump system (not the CDG reference vacuum) is shut off,

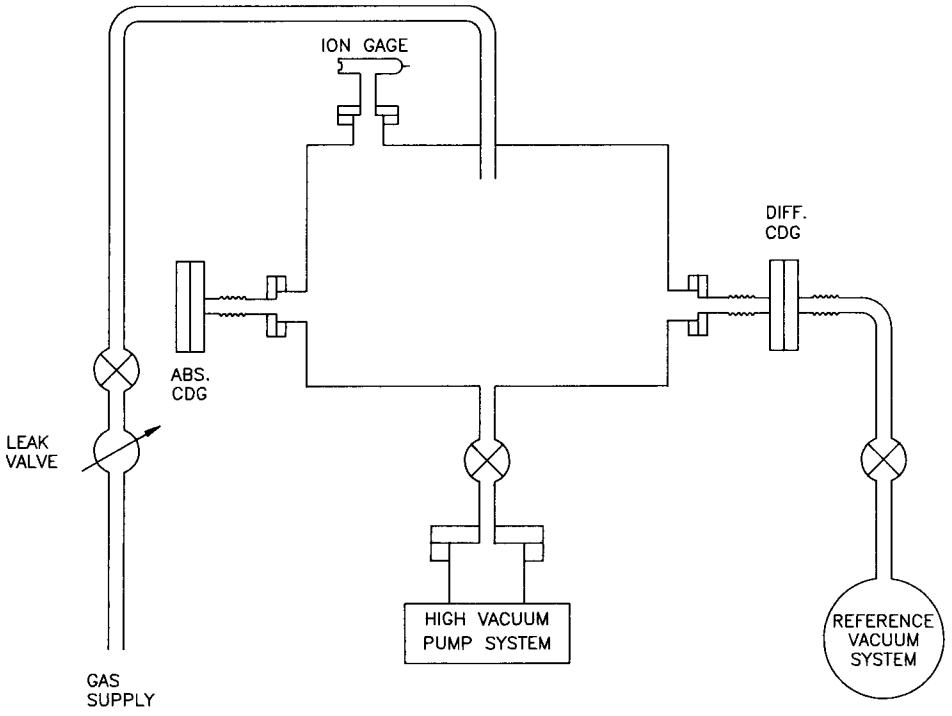


FIG. 5. System for calibrating against another CDG or transducer. This is similar to that of Fig. 7, which could, in fact, be used here. However, because the long times required for an SRG to take low pressure data are not a factor, this clean, static system should be adequate.

and gas admitted to the system to establish the desired pressure. For the lower pressures, it may be necessary to continually pump degassing byproducts while simultaneously admitting calibration gas to produce the desired pressure. At these pressures, the same arrangement suggested in Sec. VI C 4 can be used (see Fig. 7), with a calibrated CDG (the standard) substituted for the spinning rotor gage (SRG). Because the times required to obtain data are relatively short compared to those required when using an SRG, pressure changes from sorption and desorption may not be a problem, particularly for a clean system.

Use of automatic pressure control equipment is suggested to reduce stabilization times in the continuously pumped system.

3. Calibration against liquid manometers

All comments of Secs. V and VI–VI B apply.

Because of their physical simplicity, and because they produce measurements based upon fundamental physical properties, liquid manometers are often considered to be primary standards. They can be inexpensive, reliable, and can be used in pressure ranges that are of interest in vacuum work. They can cover the absolute range from the vapor pressure of the manometer liquid to several atmospheres. References 6, 7, and 24–27 describe various manometers and associated problems encountered during calibration.

The lowest absolute pressure which can be determined will be the vapor pressure of the liquid at the ambient temperature of the instrument. Commonly encountered manometer liquids include water, various oils, and mercury. Choice of manometer liquid depends upon application considerations outside of the scope of this document.

Caution: Mercury had a vapor pressure of about 0.22 Pa at room temperature. The vapor is considered to be a toxic. For this reason, use of mercury is not recommended.

Note: Some oils can be hazardous and their safety considerations are not within the scope of this document.

It is assumed that the manometer characteristics have been documented—i.e., the uncertainties of a pressure measurement arising from such factors as the measurement scale, fluid density, local gravity, tilt, temperature behavior, etc., are all understood.

In the differential mode, which generally means with a reference pressure of 1 atm, the lower differential pressure limit is, in principle, zero. However, because of random noise and accuracy requirements at a particular pressure, the low-pressure limit will not be zero. The random noise depends both on vibration and on the temperature stability of the calibration system, since gas law effects become noticeable in closed volume systems.

a. Contamination considerations. Use of a liquid manometer generally will introduce traces of the liquid into the CDG. The residual amount depends on parameters such as CDG construction, temperature, pressure, and time of exposure. Diffusion and/or condensation of the liquid vapor into the CDG ports during routine handling can occur. Care should be taken to prevent temperature gradients which can cause condensation in the CDG.

Unless some kind of clean-up procedure is used on the CDG (e.g., evacuation while heated), then when the CDG is subsequently used as a calibration device, vapor may be transferred into the gage being calibrated, and then ultimately into the final process whose pressure is being determined. The user must decide whether or not any of these factors are important.

The CDGs themselves may or may not be harmed by the vapor of the manometric fluid, depending on their construction. It is worth checking with the manufacturer. The vapor traces can be removed by pumping to below the vapor pressure at the gage temperature. Times required will be system-dependent and must be determined by the user.

b. The calibration system. Refer to Figs. 6(a) and 6(b) for possible calibration systems. All system components must be compatible with the liquid vapor.

The user must take care when operating valves not to create a pressure which will push manometer liquid out of the manometer.

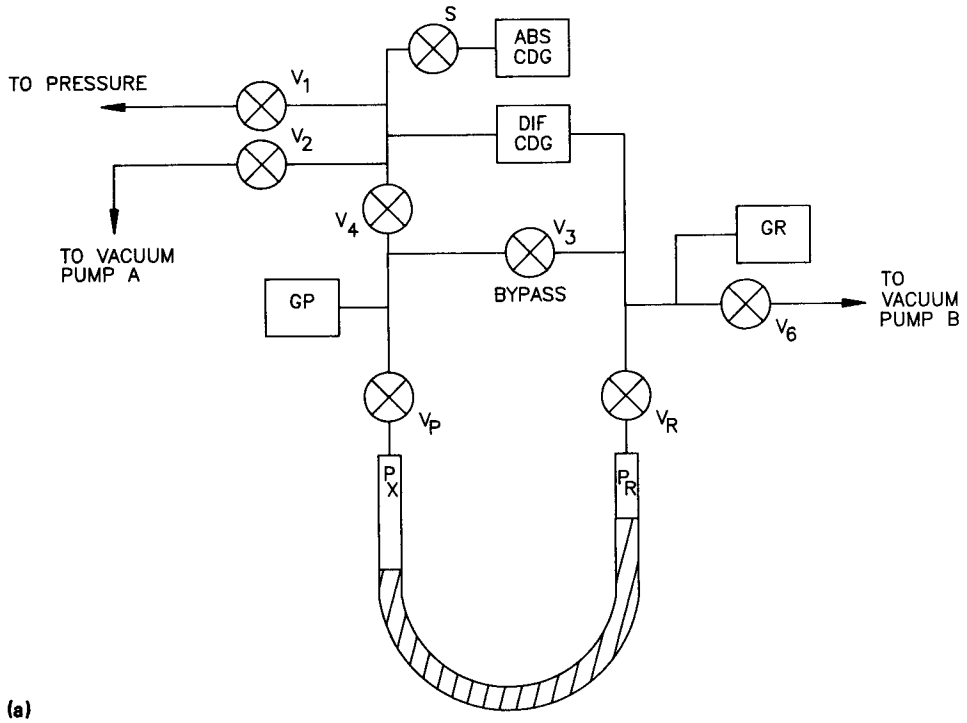
c. The calibration.

(1) Calibration of absolute CDGs or differential CDGs with pumped vacuum reference: Refer to Fig. 6(a), and note that two separate vacuum systems are recommended. Close valve V1 and isolation valve V4. Open valve S, bypass valve V3, the manometer pressure and reference side valves V_p and V_r, and slowly open vacuum valve V6. Open vacuum valve V2. Evacuate both the CDG and manometer portions of the system for a sufficient period of time so that stable zero pressures have been achieved. Gages GP and GR do not have to be calibrated, but should serve as nominal pressure indicators. Adjust the CDG indications to zero and record the indication of the manometer. Close V2, V3, and open V4. After a few minutes, the absolute CDGs should read the vapor pressure of the liquid, increased by any thermal transpiration effects which may be present. The differential CDG will read the difference between the vapor pressure of the liquid and the vacuum achieved on the reference side of the system, each of these two pressures being increased by any appropriate thermal transpiration factors. The manometer will change by an amount depending on the action of the pump on the vapor over the liquid surfaces. The change cannot exceed the liquid vapor pressure. The manometer indication will have to be arithmetically adjusted to correspond to the liquid vapor pressure if absolute CDGs are being considered, or to the difference between the liquid vapor pressure and the reference pressure indicated by the gage GR if differential CDGs are being considered.

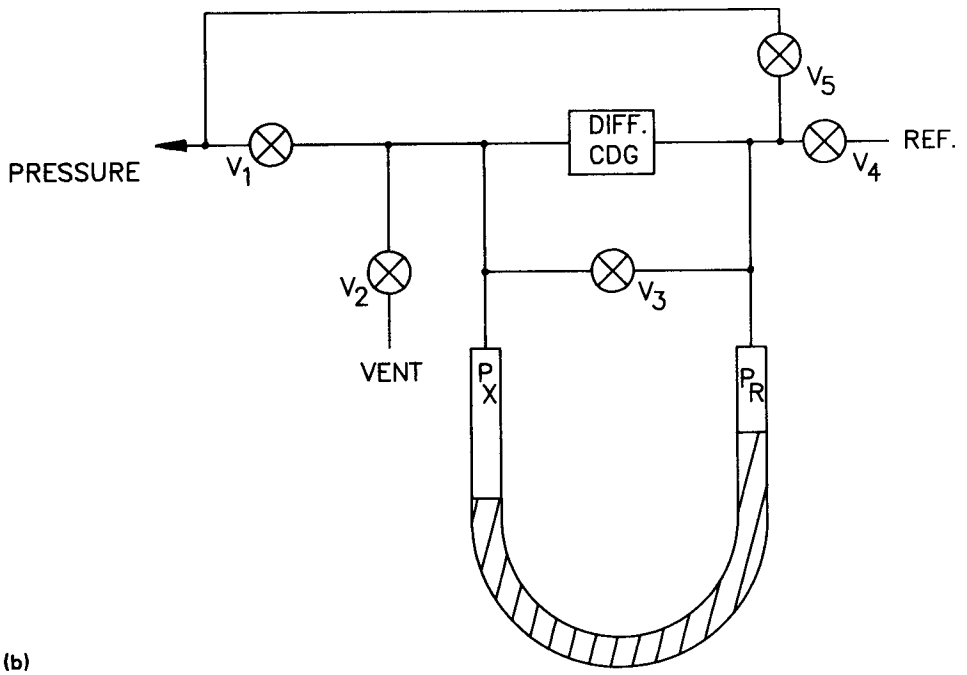
Slowly open V1 and admit gas until the desired calibration pressure is established. Read the CDG, GR, and the manometer column heights and manometer temperature. The true absolute pressure is the manometer reading corrected per the manufacturer's instruction manual.

Slowly open V1 and admit gas until the next desired calibration pressure is achieved. Repeat as above.

(2) Calibration of differential CDGs relative to atmospheric pressure: Refer to Fig. 6(b). Close pressure valve V1 and vent valve V2. Open bypass valve V3. Reference valve V4, which opens to atmosphere, should be opened, then re-



(a)



(b)

FIG. 6. (a) System for calibrating CDGs relative to vacuum against a liquid manometer. GP and GR are gages for monitoring the pressure (P_x) and reference (P_R) sides of the system. A relatively low-range absolute CDG and a relatively high range differential CDG are shown in position to be calibrated. To prevent over-ranging of the low range gage as the calibration continues on the high range gage, a shut-off valve S is used. Two independent vacuum systems are recommended. (b) System for calibrating differential CDGs relative to atmospheric pressure. The line connecting the pressure through V5 to the reference side is to prevent room air from being drawn into the system as the pressure is lowered. See the text.

closed. Zero the CDG and record the manometer zero.

Close V3 and open V4. As the liquid rises in the reference column, the gas must be able to escape. Slowly open V1 to admit gas sufficient to establish the first calibration point. Reclose V4. Allow time for equilibration, then record the CDG indication, and the manometer column heights and temperature.

Open V4, and slowly open V1. Admit gas until the next desired calibration pressure is achieved. Repeat as above.

If it is desired to keep room air out of the reference side of the calibration system as the pressure is lowered in a controlled manner (as opposed to "dumping" the pressure by opening the bypass valve V3), then the extra line and valve V5 may be added. To lower the pressure, V4 must be opened, but the retreating reference side column will cause room air to be drawn in. To prevent this, also open V5, allowing sufficient calibration gas to flow so that even as the reference column retreats, gas is flowing out of valve V4. Calibration gas is therefore being drawn into the reference column instead of room air.

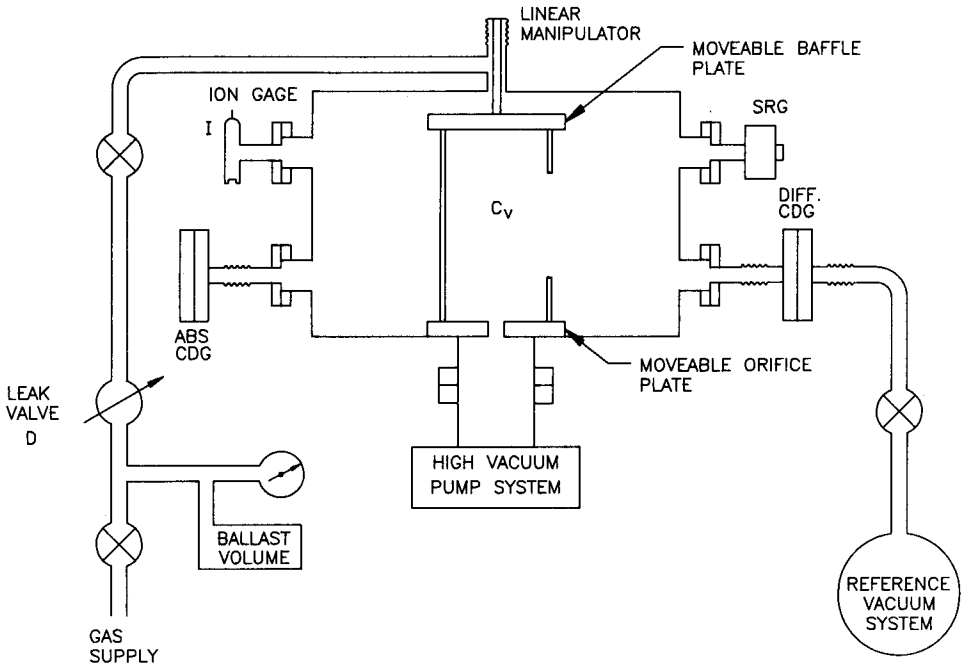


FIG. 7. System for calibrations using a SRG. It is a continuous flow system, to prevent any sorption/desorption effects from becoming a problem at low pressures. The pressure in the calibration volume C_v is maintained by gas flowing from the leak valve. The gage I need only be a crude indicator. Note the upper baffle plate and the orifice plate which can be raised to allow rapid pumpdown.

4. Calibration using spinning rotor vacuum gages (SRGs)

Note: Spinning rotor gages are also known as molecular drag gages.

All comments of Secs. V and VI–VI B apply.

The principles and details of SRG operation are beyond the scope of this document. Only a few of the more important considerations are noted. References 4, 9, and 28 deal in detail with the properties and handling of SRGs. The operational part of the instrument consists of a small (4–5 mm diam) steel ball which is magnetically suspended within a tube or thimble which is attached to the vacuum system. The ball is spun to a rotational frequency of about 400 Hz. The rate at which the frequency decreases depends on the surface roughness of the ball, the calibration gas, system pressure, temperature stability, and undesired magnetic and vibration effects, the latter collectively being called the residual drag.

This technique can cover the absolute pressure range from roughly 10^{-4}–1 Pa. The uncertainties in the SRG only are reported to be in the range of 1%–3% of reading. Uncertainties introduced by the calibration must be added to that figure. See Sec. VII C 1 b.

It is assumed that the SRG being used as the standard has an acceptable calibration, and that uncertainties associated with use on the calibration system have been documented. Each model SRG has its own set of peculiarities, some of which can cause major problems if not understood.

a. The calibration system. An appropriate system is indicated in Fig. 7.

The calibration system should be as vibration free as possible, and the SRG should be mounted in a manner which isolates it from system vibration. The SRG control head can actually be suspended or mounted in its proper position around the thimble, independent of the calibration system.

System sorption and desorption can become important factors, not only in terms of pressure change, but in terms of unknown molecular weights. The system shown uses a continuous gas flow to minimize the effect of these problems. The system should be constructed from high-vacuum components, and if base pressures below 10^{-6} Pa are required, should be bakable to temperatures of at least 100 °C. The calibration chamber C_v may be any convenient volume where the CDGs, SRG, and any auxiliary gages (such as an ion gage) all see the same pressure. Pressure uniformity within the chamber is aided by adding an upper baffle plate to scatter incoming molecules, and by making the area of the orifice exiting to the pumping system less than 1% of the chamber surface area. In the apparatus shown in Fig. 7, the upper baffle plate is connected to the orifice plate by rods. This entire unit is attached to a linear manipulator. To evacuate the chamber quickly, the unit is raised, exposing a large entrance into the pumping system. For more details, see Ref. 29.

If an ion gage is used as a base pressure indicator, it must be located so that its temperature will not affect either the SRG or the CDG.

The gas inlet goes through a variable leak valve and then into the chamber where it is directed onto the baffle plate. This variable leak valve may be of either manual or automatic operation. If valve creep is known to exist in a manual valve, it should be set for the lowest calibration pressure well before the calibration begins.

b. The calibration. All SRGs can give an incorrect reading if the temperature of the rotor is changing. A rate of temperature change of the rotor of 0.1 K/h introduces a pressure error of about 10^{-6} Pa. The lower the pressure to be mea-

sured, the more critical this becomes. Each time the rotor is spun, it warms, and it may take hours for the temperature to stabilize. If the first pressure to be measured is of the order of 10^{-3} Pa or less, it is recommended that the rotor be spun at base pressure the day before the first pressure is to be measured. The rotor should continue to spin overnight and long enough to use at the low pressure points where respinning the rotor would cause significant problems.

Be sure all proper parameters (molecular weight and viscosity of the calibration gas, temperature at which the pressure is to be measured, etc.) are entered into the SRG memory. On the day of the calibration, the SRG residual drag is determined and entered into the SRG memory. Consult manufacturer's instruction manual.

Once any amount of calibration gas is introduced into the system, it may take hours to days to reestablish the base pressure. Therefore, it is recommended that the calibration proceed from the lowest to the highest pressures.

After each pressure change, allow enough time for equilibration. This is very important at low pressures. Equilibration occurs quickly as pressures become greater than the mid-10^{-3} Pa range. When the readings for the SRG and CDGs are stable, record the readings of both devices.

Be aware that in many cases, after changing the setting of the leak valve, the valve itself may take a long time (hours) to stabilize. At very low pressures, this can be a major disruption. Above 10^{-5} Torr it probably will not matter. To avoid having to adjust this valve, a ballast volume can be used as shown in Fig. 7. The pressure in the ballast volume is raised, leaving the leak valve setting unchanged. At higher pressures the ballast pressure can be left alone and the leak valve adjusted.

Note that, at low pressures, times of the order of 5 min are required for the SRG to gather the data necessary to compute an average pressure. The CDGs being calibrated should be read at the mid-point of the data-gathering interval.

VII. DOCUMENTATION

Maintenance of documentation relating to calibrations, including procedures and results, is a matter of user policy. The following guidelines are suggested.

A. Calibration intervals

Until a history is obtained which indicates otherwise, it is wise to keep the initial calibration intervals on a device relatively close (certainly no more than a year apart). Past calibration history and interim treatment must be accounted for rather than adhering to an inflexible schedule. Comparisons at several points against a check standard (see Sec. VIII) can be used between regularly scheduled calibrations to determine if gross changes have occurred, and to determine if a scheduled complete calibration is even necessary. Regular comparisons with check standards, perhaps before each use, can serve to build a large part of the history referred to in the first sentence. Reference 29 may provide additional guidance.

B. Records

To provide objective evidence that calibration schedules are complied with and that the accuracy of each CDG is maintained, records should be kept for each CDG that is calibrated.

1. Retention

Calibration data and ancillary records should be retained for a sufficient period of time to satisfy all regulatory and contractual requirements and to allow decisions to be made to adjust the assigned calibration interval.

2. Record content

The records should include CDG identification, calibration history including any unusual observations or circumstances, necessary information required to locate each CDG in the calibration system (if applicable), and a clear indication of when the next calibration should be performed on any gage. Similar information should be kept concerning the standards against which the calibrations are to be performed.

3. Traceability documentation

If required, documentation must be available to trace the parameter values assigned to the standard to values in terms of nationally recognized standards. See Ref. 31. If a standard is calibrated by a laboratory other than the National Institute of Standards and Technology (NIST), be sure that its calibration certificate references the calibration file number and date of calibration, plus the NIST calibration file number, if applicable, and date of calibration for the standard through which NIST traceability is being claimed.

C. The calibration report

The calibration report, at a minimum, must properly identify the gage and the standard used, date of calibration, temperature of the calibration stand, calibration gas, calibration method, operator, and any special circumstances. It must also include some method for recovering the true pressure from the CDG indicated values, and contain a statement of the overall estimated uncertainty.

A table containing the indicated values of the CDG and the standard, along with the associated differences (or corrections), is a common method of presenting the data. Plots of these differences as a function of the CDG reading are highly recommended, as they provide an easy method for comparison with previous calibrations as well as a quick method for predicting corrections for pressures between the actual calibration points. The true pressure (as determined by the standard) is sometimes presented as a function of the CDG indication. The function may result from a least-squares curve fit, and is convenient for use with a computer. For low-range, heated CDGs, it is suggested that if curve fitting is used, a separate curve be fitted for the region affected by thermal transpiration.

On CDGs having range switches, large range switch-related zero shifts, if they occur, are easily handled when cor-

rections are presented in tabular form. If equations of true (as read by the standard) pressure as a function of the CDG reading are fitted to the data, it may be prudent to independently fit the data of each decade. If there are no range change offsets or if they are deemed negligible, then, depending on the pressure range covered and the accuracies required, a single fitted equation may suffice.

1. Uncertainties

The calibration report should include a statement of the estimated maximum uncertainties (accounting for both random and systematic components) associated with the indicated pressures of the CDG over the range of pressures used in the calibration, plus any extrapolated pressures. The uncertainties should include those arising from the calibration standard, the method used, and those associated with the CDG itself.

Past performance of the gage should be taken into account during the error discussion. For example, there may be predictable drifts with time which can be accounted for, or there may have been such a drastic shift from a prior calibration that its continued use under any circumstance should be questioned.

References 7, 8, 9, and 23 have information concerning the overall behavior of CDGs.

a. Assessment of uncertainties associated with the standard. References 24 and 31 describe in detail the documentation of uncertainties of particular standards. These can serve as guidelines to the methodology used to establish overall uncertainties, including uncertainties inherent to the device being calibrated.

The performance of any device and method being used as a standard should be thoroughly documented, understood, and predictable. Only if the above conditions are met can the comparison of the standard and CDG be called a calibration. Uncertainty statements from each measurement or contributing factor (temperatures, mass values, heights, etc.) must be assessed and combined with the other factors, by statistically acceptable methods, into an overall uncertainty at appropriate points within its measurement range.

If the standard is a device whose uncertainty statement itself depends on calibration, records pertaining to these calibrations must be kept in accordance with Sec. VII B.

The historical records of the standard will allow assessment of its long-term stability, which may not be initially available. Analysis of the records may indicate that a revision in the overall uncertainty statements is necessary.

Determination of the magnitudes of drifts and random behavior in the lowest useable range of the CDG should be done periodically, becoming part of the total uncertainty of the standard.

(1) Uncertainty statement for the standard: The total error budget of the standard may be expressed as a combination of the systematic and the random uncertainties. A suggested form of the statement of the estimated maximum uncertainty U is:

$$U_{\mathrm{STD}} = \text{estimated maximum random error} + \text{estimated maximum systematic error.} \quad (6)$$

For example, the uncertainty statement might read,

$$U_{\mathrm{STD}} \ (\text{in pascal}) = 10^{-2}\ \mathrm{Pa} + 10^{-3}R, \quad (7)$$

where R is the sensor reading in pascal. The 10^{-3} multiplier for R indicates a maximum systematic uncertainty of 1 part per thousand of the indicated pressure.

Note that, in this example, as the pressure goes to zero, the randomness (noise) becomes the dominant factor, while at higher pressures, the systematic uncertainties will dominate.

Note 1: All statements of Secs. VII A–VII B apply.

Note 2: The uncertainty statements given in a calibration certificate associated with the standard do not reflect the additional uncertainties introduced when using the standard to calibrate another gage. See the next section.

b. Uncertainties introduced by the calibration technique. In general, the calibration and assessment of uncertainties for the standard will have been performed in a different environment from where it will be used. The uncertainties which are to be assigned to the CDGs calibrated against the standard must therefore reflect not only the uncertainties associated with the standard and particular CDG being calibrated but also for the technique used for the calibration.

(1) Assessment: The user must identify and document the possible sources of error arising from things such as height corrections, temperature corrections or effects, pressure drops along the lines, thermal transpiration corrections, transducer orientations, operator skill (do two different operators get the same results?), knowledge of the local value of gravity, etc. A possible way to evaluate the calibration technique is to repeatedly perform calibration of the standard itself, or of an appropriate pressure measuring device which has been independently and reliably calibrated, and to document the random and systematic differences between these measurements and those given on the accompanying calibration certificate.

c. Uncertainties in the CDG being calibrated. All statements concerning assessment of uncertainties of the standard also apply here for the CDG under calibration.

The suggested statement of estimated maximum uncertainty U [Eq. (6)] becomes

$$U_{\mathrm{CDG}} = U_{\mathrm{STD}} + \text{Estimated maximum random error from the calibration technique} + \text{Estimated maximum systematic uncertainty from the calibration technique.} \quad (8)$$

2. Control charts

Control charts are graphs which give the past history of the calibrations or comparisons between standards and individual gages, usually as a function of time. As examples, they may be a series of plots of differences between the standard readings and the CDG readings, or plot of predicted values from the calibration equations, in each case along with the calibration dates. They may be used to spot trends or changes in the behavior of either the CDG or standard.

Figure 8 shows a sample control chart, comparing a CDG to some device serving as a check standard (see Sec. VIII). The devices are routinely checked at five pressures. Assume

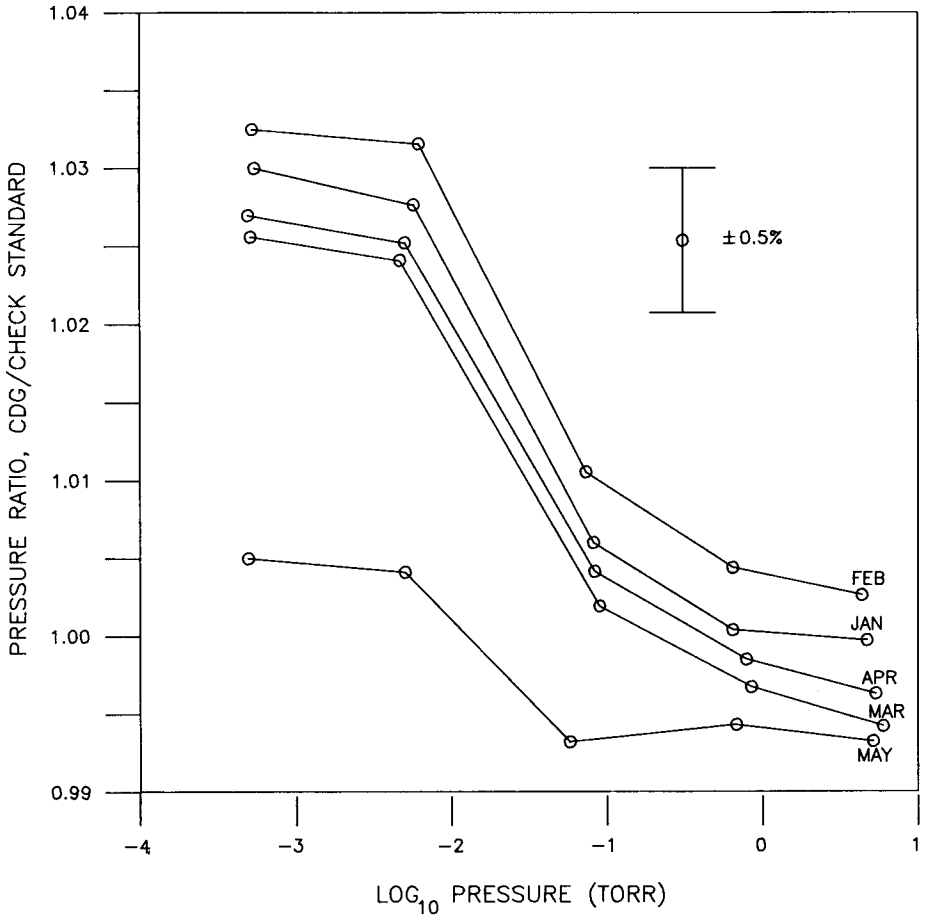


FIG. 8. Sample control chart, showing the ratio of a particular CDG to the check standard as a function of pressure. The check standard need not be accurate, but should be stable. Assume the facility requirement is that the curves remain within $\pm 0.5\%$ of the original. From January through April, this requirement is satisfied. Then, in May, a shift of the order of 2% occurred for the lower 3 points, and no points meet the requirement of being within $\pm 0.5\%$ of the original (January) calibration (See Sec. VII C 2). This last would require recalibration of the CDG.

the facility policy is that for shifts within $\pm 0.5\%$ of the original comparison, no recalibration is required. This condition is met for the January through April calibrations. But the May calibration shows a 2% shift. This requires recalibration of the standard. Reference 32 may provide further guidance.

D. Labeling practice

In order to provide a visual means of alerting the user to its calibration status, each calibrated CDG should be labeled in a prominent place.

The label should therefore show the data of the last calibration and who (or which facility) performed the calibration, as well as the scheduled recalibration date.

VIII. CHECK STANDARDS

A check standard is a device which may or may not be fully calibrated but which is known to be stable. It is not necessarily the same as a transfer standard which can be used to calibrate other devices. It is used to make occasional comparisons at several pressure points simply to confirm that the device being checked (in particular, a working standard) does or does not need a complete calibration.

The check standard may be used as often as desired to compare several points with either a working or primary standard, with the first comparison having been done as soon as possible after a newly calibrated standard became available. After each comparison, the data are examined to see if any changes have occurred which are beyond some limit acceptable within the particular laboratory. If no such change is observed, then either the standard and check standard have changed in the same way, or neither the standard of check standard has changed (within the acceptable limit). Failure to detect drifts because of the former situation would become less likely if more than one check standard is used either simultaneously or during alternate checks. If a larger than acceptable change has occurred, and no independent means is available to determine which of the two devices has shifted, then the standard should be recalibrated, even though it may have been the check standard which changed.

Note: The important factor is changes, not absolute accuracies.

The method of comparison is arbitrary, and depending on the range may be similar to any of those discussed in this document. However, it should be essentially the same each time.

It is recommended that the check standard be left evacuated and with power on when not in use.

Checking intervals should be a documented part of the laboratory procedures.

Note: If the plots, or whatever comparison records are kept between the standard and check standard indicate that

the random portion of the uncertainty is, say, 0.1% of reading over time periods on the order of a year, then this can become part of the uncertainty statement of the CDG. A higher level calibration laboratory which may see the CDG once every two years might be forced to assign a much higher uncertainty, based on its experience with a large sample of similar devices. This necessarily penalizes stable CDGs and assigns unstable ones better than actual behavior. If a laboratory has such documentation, it is perfectly acceptable to make use of it.

DISCLAIMER: This Recommended Practice is based on sources and information believed to be reliable, but the American Vacuum Society and the authors disclaim any and all liability whatsoever relating to the techniques and procedures outlined in this Recommended Practice. Furthermore, The American Vacuum Society and the authors disclaim any warranty, safety, or the result of the procedures or other applications outlined herein. The American Vacuum Society (AVS) does not endorse any products, processes, manufacturers, or suppliers. Nothing in this document should be interpreted as implying such an endorsement. Safety considerations are the responsibility of the user of this document.

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This bibliography is not intended to be exhaustive. Rather, the intent is to present complementary readings for this document.

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X. APPENDIX

This appendix contains more detailed explanations of some of the sections discussed above. The order and section designations correspond to those of the main text.

A. Section III: Terminology

The *Dictionary of Terms*, Ref. 1, contains many definitions appropriate for this document. Below are several additional definitions for the purposes of this document, which may expand or modify other corresponding definitions.

Absolute pressure: The force per unit area molecules exert on a surface.

Base pressure: The lowest absolute pressure achievable in a given system.

Calibration factor: A factor by which an observed parameter must be multiplied to convert the parameter into a true (as defined by some standard) value. This factor may not be constant over the range of interest.

Error: The algebraic difference between the observed and true value (observed-true) of a property or parameter.

Manometer: A pressure measuring device which depends on the displacement of a liquid column. The pressure is calculated from the product of the liquid density, local gravity, and the height of the displaced liquid column.

Reference pressure: The pressure on the reference side of a differential device. It may or may not be the system base pressure.

Sensor: A device which converts pressure to capacitance.

Transducer: A device converting the parameter or property being sensed or measured into a convenient form for analysis. In this document, it comprises a sensor plus electronics which translates pressure into an analogous electrical signal.

Transfer standard: Any measurement device can be a transfer standard, used to transfer "true" values of a property or parameter from itself to another device. However, the word standard implies that long- and short-term stabilities and sources of random and systematic uncertainties have been investigated, are understood, and are satisfactory for the desired transfer.

B. Section V A: The calibration system

Outside of a standards-level calibration laboratory and in general field calibrations, it is recognized that conditions are often less than perfect. The users in this situation can only be cautioned that it is critical to perform a rigorous, realistic error analysis, complete with documented results of comparisons of CDGs in the calibration lab and in the field, in order to have confidence in the process and final result.

Often gages which come back into the calibration laboratory have been exposed to situations which have loaded them with materials which may in turn wreak havoc on a clean calibration system. The lab operator should be aware of the prior use of the CDG before connecting it to the calibration system. It may be necessary in some cases to have an auxiliary pumping stand to which a potentially dirty CDG can be connected for removal of volatiles by pumping until an acceptable residual pressure level is achieved. (The acceptable pressure may well depend on the contaminant.) Sometimes heating to the maximum rated operating temperature of the CDG can assist in the removal of volatiles. Note that the pumping away of undesirable contaminants can be applied to differential units not ordinarily used under vacuum, as well as to absolute gages.

C. Section V C: Initial adjustments

There is a pervasive notion that any instrument which comes in to a calibration laboratory must be adjusted to "within specifications", whatever that may mean, before it is used. However, the past calibration history can be made irrelevant by readjusting the electronics, including changing the span (sensitivity) setting. (This does not refer to the routine adjustments for such as nulls, full scale settings, and transducer zeros.) Therefore, a calibration should be performed before any internal electronic adjustments are made.

Obviously if an instrument is so far out of adjustment as to make it unusable, alignment procedures must be undertaken. In general, however, this is not a good idea. The purpose of the calibration is to provide corrections to the readings. In general, the magnitudes of these corrections are of no consequence; therefore readjusting the electronics to make these corrections small is not necessary.

It is recognized that at the field-user end of the calibration chain, the operator may not want to be bothered with applying corrections. It is assumed that instrument indication is correct, to within the stated uncertainties, as it is read. In this situation, where the instrument is to be made as direct reading as possible, it is recommended the calibrating laboratory maintain the calibration history by doing an "as received" calibration followed by a "set into specs" calibration.

D. Section V D 1: Signal conditioner zeroing

Generally, one need not be concerned about nonlinearities in the electronics between zero and full scale. The effects of these become part of the calibration. If the nonlinearities are gross enough to require adjustments, be sure that such changes are documented. See Sec. VII.

E. Section V F 2: Thermal transpiration

Because of thermal transpiration, the temperature and species of the gas being measured and the temperature of the transducer should appear on every calibration report. This will permit a user or calibrator who is working with a different set of conditions to make appropriate corrections to observations.

Note that Eq. (4) of Ref. 10 contains an inverted conversion factor.

Refer to Fig. 9(a), which shows the ratio of the pressure as seen by a CDG whose calibration factor, other than ther-

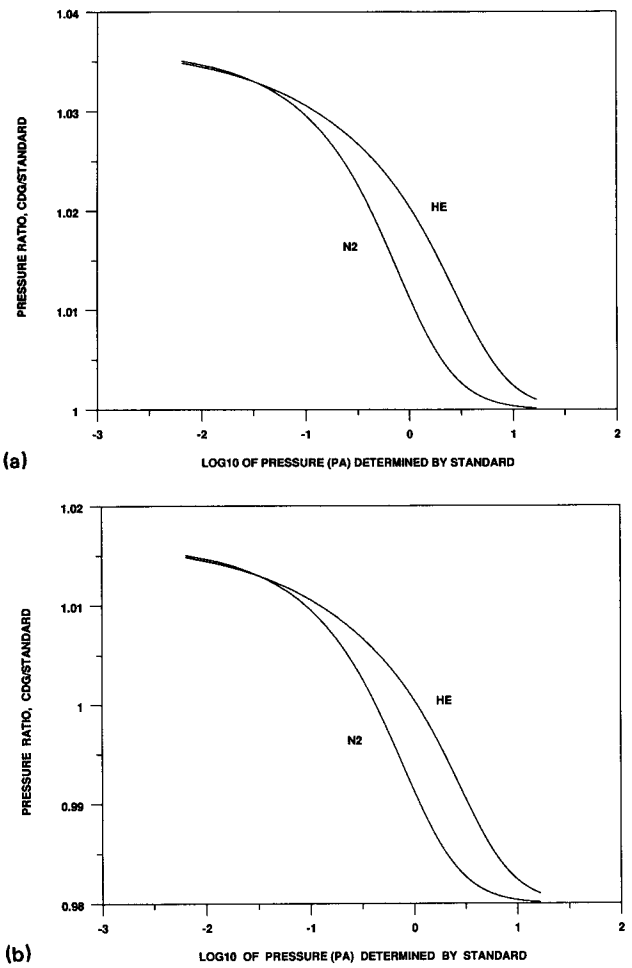

FIG. 9. (a) Typical plot of the ratio of the pressure indication of a heated (45 °C) transducer to the pressure determined by of a standard maintained at room temperature (23 °C) as a function of the log of pressure indication of the standard. The plot assumes that the transducer is perfect, that is, beyond the thermal transpiration region, the calibration factor is "1". Curves are shown for both He and N_2, indicating the effect of molecular species on thermal transpiration. The internal diameter of the tubing connecting the hot and cold volumes is assumed to be 4.76 mm. (b) Same as (a), only here the transducer is assumed to have an upperend calibration factor of 0.98. Note the shift of the entire curve. This must be accounted for when thermal transpiration calculations are being made.

mal transpiration effects, is unity. It is operating at 45 °C, and system to which it is connected operating at 23 °C. As the pressure decreases, the pressure within the CDG becomes greater than that in the system. As the mean free path of the gas approaches the dimensions of the interconnecting tubing, the pressure indicated by the CDG approaches a limit which will be higher than that in the system to which it is connected by a factor which is the square root of the ratio of the absolute temperatures of the CDG to that of the system, i.e.,

$$\text{Limiting thermal transpiration ratio} = \text{TTR} = P_{\text{CDG}}/P_{\text{System}}\,(\text{at very low pressure}) = [(273.15 + t, \text{°C of CDG})/(273.15 + t, \text{°C of system})]^{1/2}, \quad \text{(A1)}$$

where the temperatures are in degrees Celsius.

For a given system and temperature difference, the shape of the curve which describes the ratio of pressures in the CDG to that in the system, as a function of pressure, is dependent on the molecular species of the gas. However, the TTR limit of Eq. (A1) will be the same for all gases. Thus, if a calibration is performed with helium and the transducer is subsequently used with nitrogen, errors of the order of one percent in the measured CDG pressure will occur between 2 and 12 Pa [see Figs. 9(a), 9(b)]. Even larger differences will occur for other gases. Users of heated CDGs in the region between 0.1 and 100 Pa should be very much aware of this property when using gases other than that used for the CDG calibration.

Note: If the calibration factor is not unity, then the low-pressure thermal transpiration limit of Eq. (A1) must be corrected by the same percentage that the calibration factor at pressures above the thermal transpiration region differs from unity.

Example: Refer to Fig. 9(b). Assume a pressure standard with no thermal transpiration effects is operating on a system. Suppose the reciprocal of the calibration factor, as determined by the ratio of the CDG reading P_{CDG} to the pressure determined by the standard P_{STD} near the upper end of the range of the transducer is 0.98, i.e.,

$$P_{\text{CDG}}/P_{\text{STD}} = 0.98. \quad \text{(A2)}$$

As seen in the figure, the entire curve predicted by the equations which describe thermal transpiration (to be discussed below) is shifted by 2%, the CDG indication being too low. This means that the limiting thermal transpiration ratio TTR is also too low by 2%. Thus for this transducer, the correct limiting TTR should be the limiting thermal transpiration ratio [Eq. (A1)] multiplied by 0.98,

$$\text{TTR}_c = \text{TTR} \times 0.98. \quad \text{(A3)}$$

In the absence of any other method for setting the transducer zero (suppose for some reason the system base pressure is of the order of 0.1 Pa, which would prevent the proper zeroing of many CDGs), the above equations can be used to obtain a reasonable approximation to the zero offset, assuming the pressure in the system to which the CDG is connected is known by some independent means. Use the two temperatures to determine the TTR. At pressures below approximately 1 mTorr, offset the CDG indication of pressure (either physically or arithmetically) so that Eq. (A1) is satisfied. Next complete the calibration procedure and determine the upper end ratio as required by Eq. (A2). This permits the second adjustment to the zero offset through Eq. (A3), and the calibration can now be completed.

The following is taken directly from Ref. 14. It gives the general details of the calculation of the thermal transpiration effect. The equation numbers are the same as in Ref. 14. Several parenthetical comments { } have been added.

"A modified form of the Liang equation was proposed by Takaishi and Sensui {Ref. 33} based on the measurement of thermal transpiration at temperatures closer to 300 °C. Their equation has the following form:

$$\frac{P_2}{P_1} = \frac{AX^2 + BX + CX^{1/2} + (T_2/T_1)^{1/2}}{AX^2 + BX + CX^{1/2} + 1}, \quad (8)$$

TABLE 2. Values of the constants A^*, B^*, and C^*.

Gas	A^*	B^*	C^*
He	$1.50\times E5$	$1.15\times E2$	19.0
N_2	$1.20\times E6$	$1.00\times E3$	14.0
Ar	$1.08\times E6$	$8.08\times E2$	15.6
SF_6	$1.53\times E7$	$2.42\times E4$	4.4

where

$$A = A^*(T^*)^{-2}, \tag{9}$$

$$B = B^*(T^*)^{-1}, \tag{10}$$

$$C = C^*(T^*)^{-0.5}, \tag{11}$$

$$T^* = 0.5(T_1 + T_2), \tag{12}$$

and

$$X = (0.133)^{-1}P_2 d, \tag{4}$$

{where X has units of Pascal meter, and d is the internal diameter of the tubing where the temperature transition takes place. The original paper incorrectly omits the -1 exponent in Eq. (4).}

"The values of the reduced constants A^*, B^*, and C^*, i.e., the constants with the temperature dependence removed, for the gases H_2, Ne, Ar, Kr, CH_4, Xe, He, N_2, and O_2 were found by Takaishi and Sensui by fitting either their experimental data, or data already available in the literature, to Eq. (8) {above}. The values they obtained for the gases used in this study are shown in Table 2.

"Where Takaishi and Sensui give only the range in which the constant lies, then the mean of the range is given in the table."

"From their results Takaishi and Sensui derived the following simple empirical equations relating the constants A^*, B^*, and C^* to the molecular diameter (D) of the gas. These equations can be written

$$A^* = 1.4\times 10^4 \exp(1.17D \times 10^{10}), \tag{13}$$

$$B^* = 5.6\times \exp(1.40\times D \times 10^{10}), \tag{14}$$

$$C^* = (1.10\times 10^{-8}/D) - 14, \tag{15}$$

where D is the molecular diameter in meters.

"The value of the molecular diameter is available for a wide range of gases and even for those gases for which it is not, it can be derived from viscosity data using the kinetic energy expression

$$\eta = (5/16D^2)(mKT/\pi)^{1/2}. \tag{16''}$$

This ends the excerpt from Ref. 14.

If the CDG is calibrated under one set of conditions (i.e., gas, temperatures, connecting tubing size) and then used under different conditions, the correction for the effect of thermal transpiration may be made as follows.

For the initial calibration, Eq. (8), above, applies. Rewrite it as

$$P_2/P_1 = P_c/P_s = f, \tag{A4}$$

where the subscripts c and s refer to the indications of the CDG and standard. The standard, in turn, predicts the true pressure in the calibration system. The right-hand side of Eq. (8) is represented by f.

Under different conditions,

$$P'_c/P'_s = f'. \tag{A5}$$

If the CDG reading is the same in both cases, one obtains

$$P_s f/f' = P'_s. \tag{A6}$$

The procedure is to first calibrate and obtain a relationship f between the CDG reading and the standard. If enough data are taken in the thermal transpiration region, this may be in the form of a least-squares fitted equation, which takes the place of Eq. (8). Otherwise Eq. (8) can be used as a reasonable approximation. When using the CDG in another set-up, the correct pressure is obtained in two steps. First, the original calibration data are used to obtain what would be the correct pressure under the same conditions as were used for the calibration. Equation (8) is then used to predict the value of f' for the new conditions. The correct pressure under the new conditions is then computed from Eq. (A6).

Note: If identical transducers with nominally the same calibration factors are placed on a calibration system and intercompared, the following situation arises. For the first transducer, $P_{c1}/P_s = f$ and for the second, P_{c2}/P_s also (approximately) $= f$. If $c2$ is designated as a standard, then P_{c1}/P_{c2} approximately $= 1$ at all pressures, and there is no apparent thermal transpiration effect. Of course it is still present in both transducers, and Eq. (A4) must still be used to obtain the pressure in the calibration system.

F. Section VI C 6: Calibrations of differential CDGs relative to atmospheric pressure

Problems may arise as the atmospheric pressure varies. If for example a liquid manometer is being used as the standard, once the liquid begins oscillating in response to an atmospheric pressure change (perhaps from someone slamming a door somewhere), it takes time to stop. At any instant, the manometer may give an apparent reading which differs from the pressure indicated by the CDG. This effect will be random, and, depending on the accuracy required, may be overcome by taking many repeated readings at or near the same pressure.

In the case where a liquid manometer is the standard, with its reference side connected in parallel with the reference side of the device being calibrated, be aware that as the applied pressure is increased, the reference side gas is compressed unless it is vented to atmosphere [see Fig. 6(b)]. Therefore the reference pressure may have to be adjusted.